

# PATENT SPECIFICATION

(11) 1 202 961

## DRAWINGS ATTACHED

1 202 961

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## (54) TUBE PRODUCTION

(71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1., a British Company do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the production of tubing of plastics and in particular to the production of tubing of high melting polymers such as 4-methyl pentene-1 polymers.

In British Patents 942,297; 968,935; 1,001,801; 1,014,866 and 1,085,914 we have described a series of polymers and copolymers based on 4-methyl pentene-1. These polymers combine a good chemical resistance, high light transmission (in many cases in excess of 90% when measured by A.S.T.M. Test 1746-62T) and a high melting point (usually somewhat above 240°C). These properties make 4-methyl pentene-1 polymers suitable for use as connector pieces or tubes in laboratory, hospital and dairy equipment, which can be sterilized. In order to take advantage of the good properties of these polymers, however, it is necessary to produce tubing having a smooth surface finish.

According to the present invention there is provided a method of the production of tubing of a synthetic thermoplastic polymeric material wherein an extruded parison of the polymeric material is passed, in sequence through a vacuum sizing die and then through a contraction die (as defined herein).

This procedure has been found to be particularly suitable for the production of tubing formed of 4-methyl pentene-1 polymers. Difficulty is experienced in the extrusion of unsupported tubing of large diameter (diameter in excess of about 4

inch) due to the low melt viscosity of 4-methyl pentene-1 polymers. If a sizing die with internal air pressure is used, the resulting product is unsatisfactory due to a tendency for the die to block and produce solid rod.

According to a further aspect of the invention there is provided apparatus for the production of tubing of a synthetic thermoplastic polymeric material comprising in combination, a vacuum sizing die and a contraction die (as defined herein) connected together to provide a continuous unbroken passageway therethrough.

The assembly is conveniently immersed in a water bath whereby the tube passing from the contraction die is completely immersed in the water bath and cooled.

The method and apparatus of the present invention are particularly suitable for the production of tubing of 4-methyl pentene-1 polymer including tubing of diameter in excess of  $\frac{1}{4}$  inch.

The term "4-methyl pentene-1 polymer" is used herein to include both homopolymers and copolymers of 4-methyl pentene-1, and in particular polymers such as those described in the aforementioned British patents.

By a "contraction die" we mean a die having a passageway therethrough the diameter of said passageway decreasing in passing from the entry point, there optionally being a parallel portion adjacent to the exit point. The contraction die permits the tube to contract as it cools on passing from the vacuum sizing die and prevents water from the water bath from passing into the sizing die thereby causing the external surface of the tube to be irregular. The parallel portion serves to polish the outside diameter of the tube. The taper in the contraction die is dependent both on the diameter of the tube being formed and also on the

material from which the tube is formed, for example, for a one inch diameter tube of 4-methyl pentene-1 polymer, the taper is about 0.010 inches.

5 In order to more fully describe the present invention, reference is made, by way of example, to the drawings accompanying the provisional specification wherein:

Figure 1 is a cross-section of a vacuum  
10 sizing die;

Figure 2 is a cross-section of a contraction die; and

Figure 3 is a cross-section of the combination of vacuum sizing die and contraction  
15 die, assembled ready for use.

The vacuum sizing die has a smooth, polished central bore 1, one end of which is counterbored to form a recess 2 into which one end of the contraction die is fitted. The  
20 body of the sizing die is divided into three zones by the end walls 3 and 4 and inner partitions 5 and 6. The two outer zones 7 and 8 are vacuum zones, whilst the inner zone 9 is a cooling zone. The zones 7 and 8  
25 are connected to each other by tubes 10 and 11 which pass through the cooling zone 9. Into the outer perimeter wall 12 of the sizing die are fitted connector pipes 13, 14 and 15. Pipe 13 connects into zone 8 and provides  
30 the vacuum for zones 7 and 8. Pipes 14 and 15 connect into zone 9 and are the inlet and outlet for cooling water. Two double rings of suction holes 16 and 17 are provided near each end of the bore 1 and connect with  
35 zones 7 and 8 respectively.

The sizing die is conveniently secured directly to the end of the water bath which is represented by flange 18. The sizing die is secured to flange 18 by means of bolts (not  
40 shown) passing through threaded bolt holes such as 19 and 20 in flange 18 and end wall 3 respectively.

The contraction die comprises a tubular piece 21 having a flange 22 near to one end.  
45 The internal bore 23 of the contraction die decreases in diameter in passing from the flanged end of the die, a final portion 24 at the exit being parallel. A series of threaded bolt holes 25 are provided in the flange 22.

50 As shown in Figure 3, the bore 1 of the sizing die and the bore 23 of the contraction die should be accurately aligned and there should be no gap at the junction between the two bores. The two dies are secured together by bolts (not shown) passing through  
55 the bolt holes 25 and a corresponding series of bolt holes (not shown) in the end wall 4. The bores are polished radially, not in the line of draw, to prevent sticking.

60 In operation the whole assembly shown in Figure 3 is located in a water bath, which is initially empty. The pipe 13 is connected to a vacuum pump, a water pump has been found to be adequate, and a low pressure  
65 can thus be established in zone 8, and,

through tubes 10 and 11, in zone 7. Cooling water is passed into zone 9 through pipe 14 and removed through pipe 15.

Hot extruded parison at a temperature in excess of the melting point of the plastic, for  
70 example, with 4-methyl pentene-1 polymer, the tubing is at a temperature of about 275°C, is passed into the bore 1 of the vacuum sizing die. The suction through the holes 16 and 17 causes the tubing to con-  
75 form closely with the surface of the bore 1 and thereby attain a good surface finish. The tubing cools and contracts in passing through the contraction die and then passes into the bath. Once sufficient tubing has  
80 been formed to pass the length of the die, the bath may then be filled with sufficient water to cover the tubing and the contraction die.

The method and apparatus described  
85 herein are particularly suitable for the production of tubing of 4-methyl pentene-1 polymer of good quality having a good surface finish. In making tubing of 4-methyl pentene-1 polymer using the described  
90 method, it is convenient to adjust conditions to provide some drawdown, for example 10-15%.

#### WHAT WE CLAIM IS:—

1. A method for the production of tubing  
95 of a synthetic thermoplastic polymeric material wherein an extruded parison of the polymeric material is passed through a vacuum sizing die and then through a contraction die (as defined herein). 100
2. A method according to claim 1 wherein the polymeric material is a polymer of 4-methyl pentene-1.
3. A method according to claim 2 wherein the tubing formed has a diameter in excess of 0.25 inches. 105
4. A method according to claim 2 or 3 wherein the tubing is at a temperature of about 275°C.
5. A method according to any of claims  
110 1 to 4 wherein the tubing is passed from the contraction die into a water bath.
6. A method according to any of claims 1 to 5 wherein conditions are adjusted to provide drawdown. 115
7. A method as claimed in claim 1 and substantially as hereinbefore described with reference to the drawings accompanying the provisional specification.
8. Apparatus for the production of tubing  
120 of a synthetic thermoplastic polymeric material comprising a vacuum sizing die and a contraction die (as defined herein) connected together to provide a continuous unbroken passageway therethrough. 125
9. Apparatus according to claim 8 wherein the passageway through the contraction die is provided with a parallel portion adjacent the exit point.
10. Apparatus according to claim 8 or 9 130

wherein a cooling jacket is provided in the middle portion of the passageway through the vacuum sizing die.

11. Apparatus according to claim 10  
5 wherein suction holes are provided near each of the passageway through the vacuum sizing die.

12. Apparatus according to claim 11  
10 wherein the suction holes are arranged as a double ring at each end of the vacuum sizing die.

13. Apparatus according to any of claims 8 to 12 wherein the surface of the passageway through the sizing die and the  
15 contraction die has been polished radially.

14. Apparatus according to any of claims 8 to 13 wherein the sizing die is secured directly to the end of a water bath.

15. Apparatus as claimed in claim 8 and substantially as hereinbefore described with 20 reference to the drawings accompanying the provisional specification.

16. Tubing of a synthetic thermoplastic polymeric material whenever produced by the method of any of claims 1 to 7. 25

17. Tubing of a polymer of 4-methyl pentene-1 whenever produced by the method

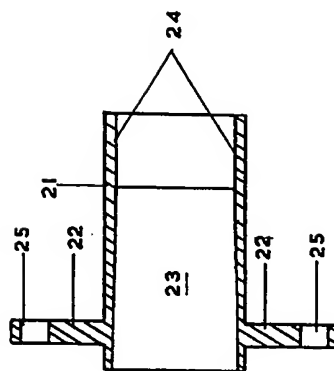
D. G. JAMES.

of any of claims 1 to 7.

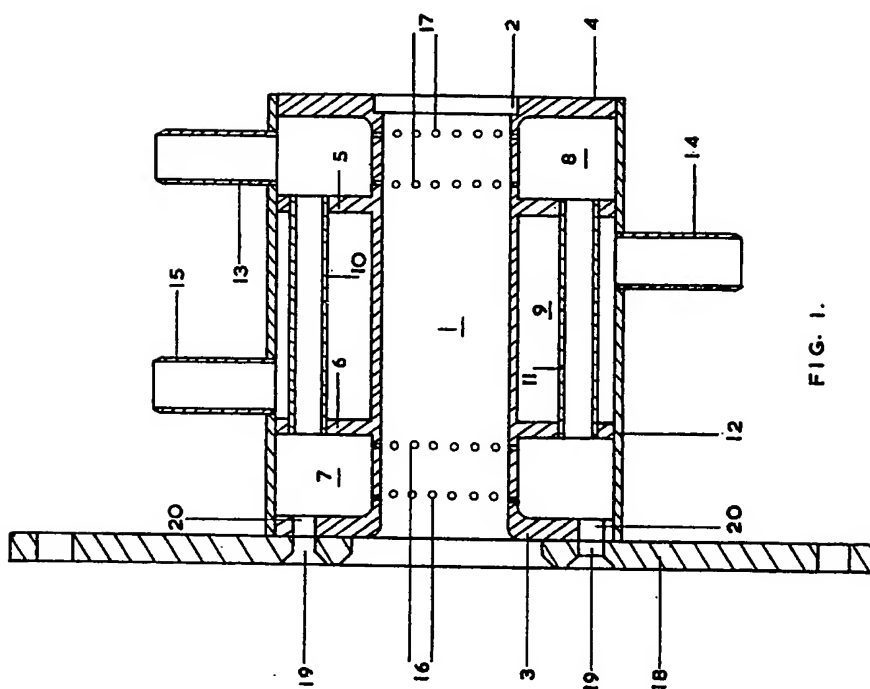
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**2 SHEETS**

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the Original on a reduced scale.  
SHEET 1



**FIG. 2.**



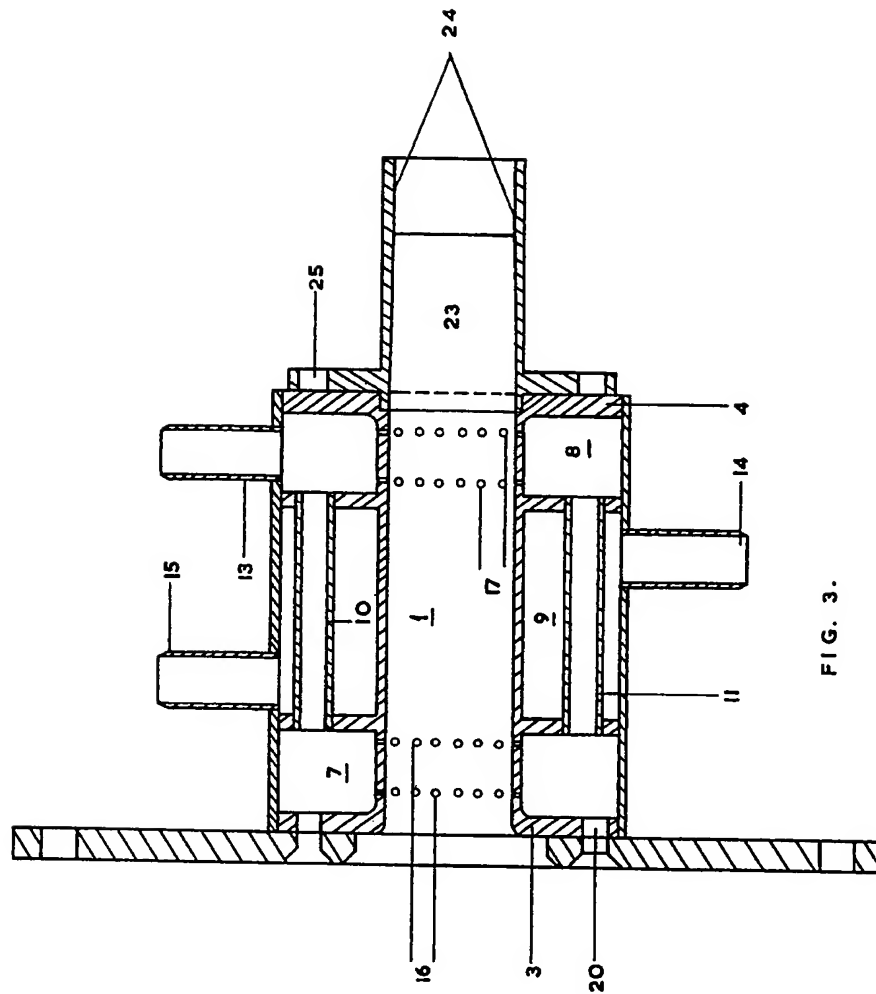
**FIG. 1.**

**2 SHEETS**

## PROVISIONAL SPECIFICATION

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**SHEET 2**



**FIG. 3.**